



JOURNAL OF THE AMERICAN CHEMICAL SOCIETY

J. Am. Chem. Soc., 1997, 119(7), 1599-1608, DOI: [10.1021/ja961883q](https://doi.org/10.1021/ja961883q)

Terms & Conditions

Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not otherwise permitted to reproduce, republish, redistribute, or sell any Supporting Information from the ACS website, either in whole or in part, in either machine-readable form or any other form without permission from the American Chemical Society. For permission to reproduce, republish and redistribute this material, requesters must process their own requests via the RightsLink permission system. Information about how to use the RightsLink permission system can be found at <http://pubs.acs.org/page/copyright/permissions.html>

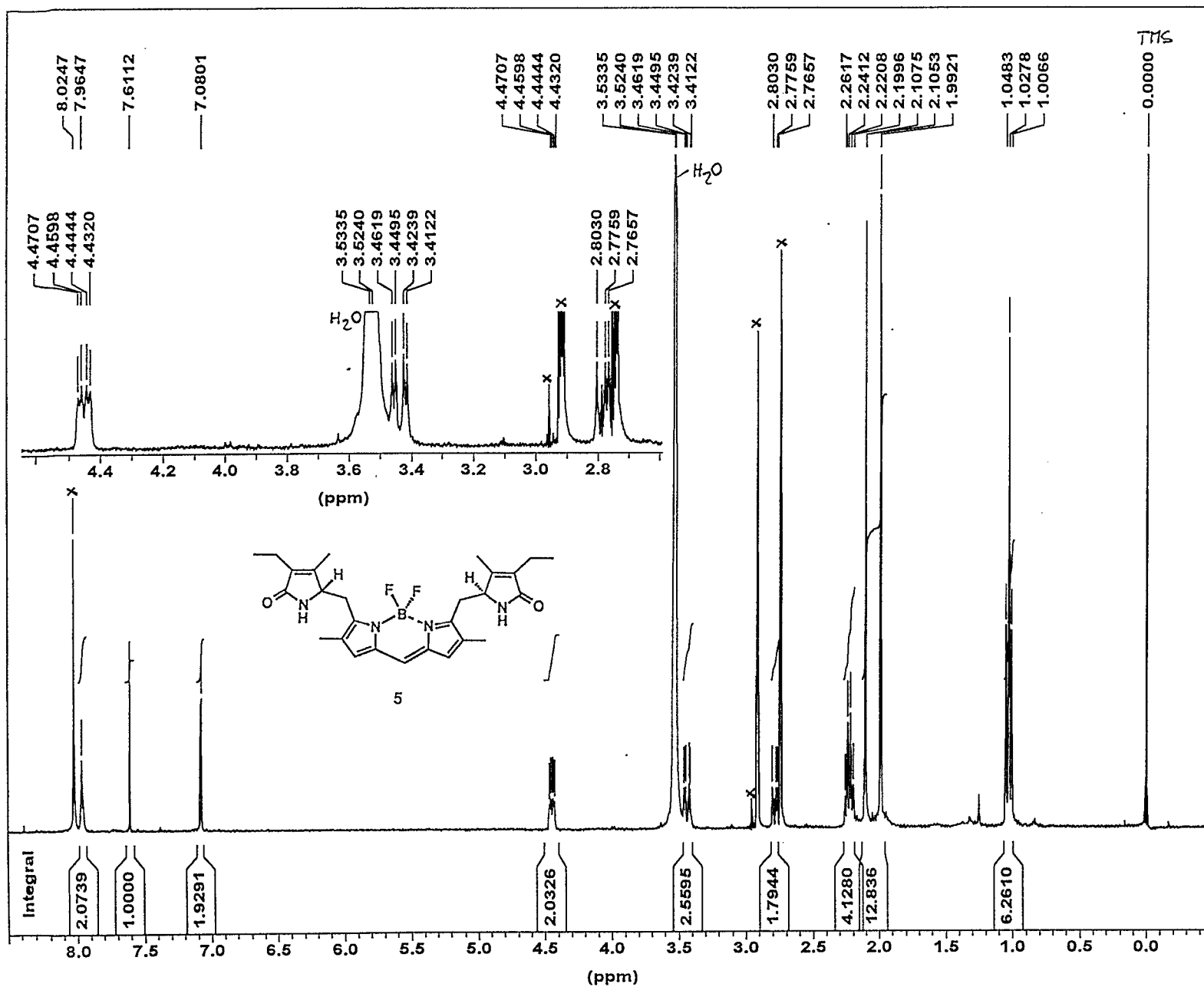


ACS Publications

MOST TRUSTED. MOST CITED. MOST READ.

Copyright © 1997 American Chemical Society

Compound 5: ¹H-NMR (360.14 MHz) in DMF-d₇



*** Current Data Parameters ***

NAME : fehr8912

EXPNO : 1

PROCNO : 1

*** Acquisition Parameters ***

AQ_mod : qseq

AUNM : NOEMULT.AU

BF1 : 360.1300000 MHz

FW : 5400.00 Hz

INSTRUM : AM

NS : 3018

O1 : 7404.12 Hz

O2 : 5500.00 Hz

SFO1 : 360.1374041 MHz

SW : 11.9687 ppm

SW_h : 4310.345 Hz

TD : 32768

TE : 298 K

*** Processing Parameters ***

FT_mod : fqr

OFFSET : 11.159 ppm

SF : 360.1355407 MHz

SI : 16384

*** 1D NMR Plot Parameters ***

SR : 5540.70 Hz

AQ_time : 3.8010680 sec

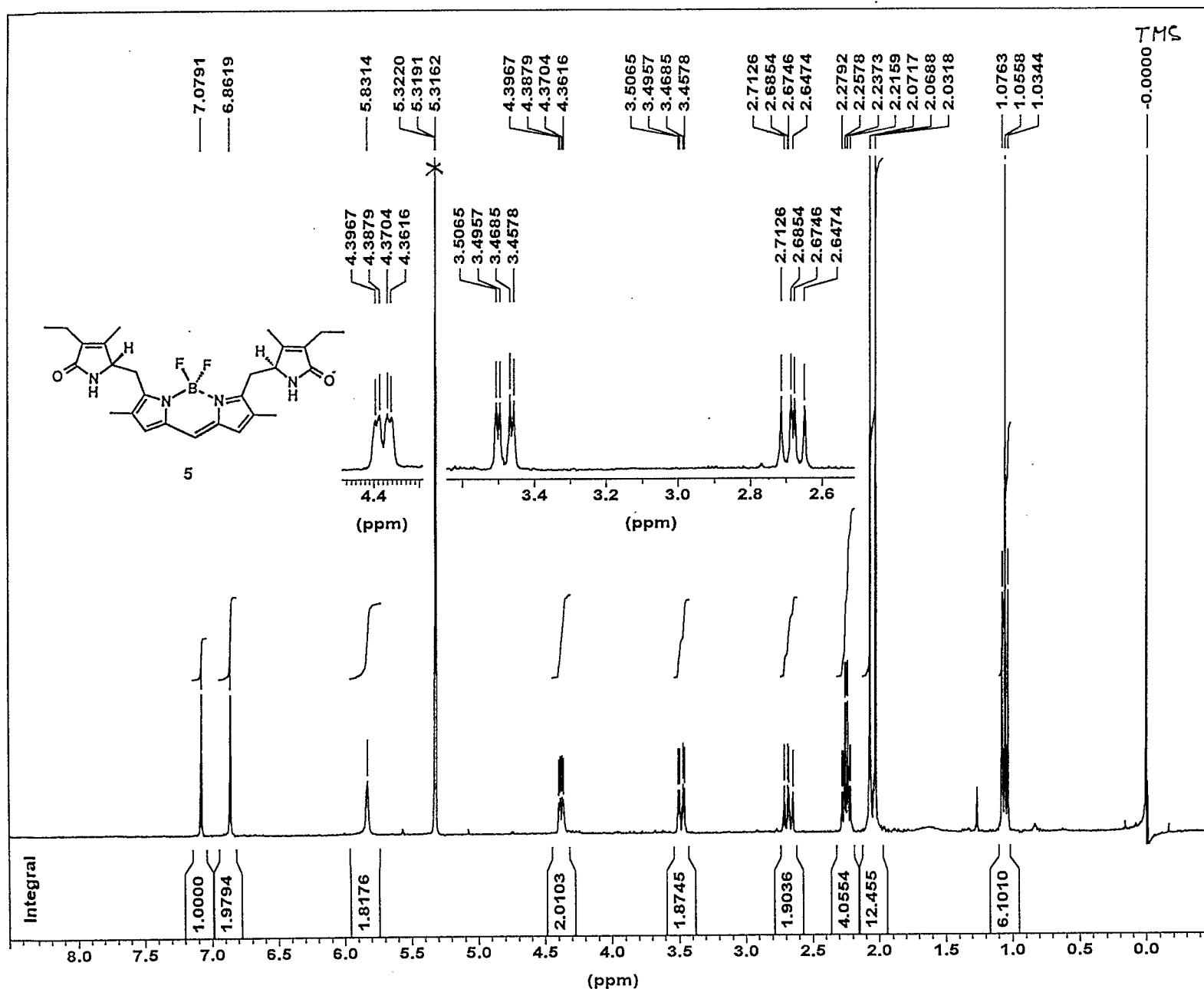
x ... Lösungsmittelpeak

¹H-NMR (AM360)

Probe: S,S-Urobilin als BF₂-Komplex

Lösungsm.: CD₂Cl₂ (TMS)

Compound 5: ¹H-NMR (360.14 MHz) in CH₂Cl₂



*** Current Data Parameters ***

NAME : fehr7568

EXPNO : 1

PROCNO : 1

*** Acquisition Parameters ***

AQ_mod : qseq

AUNM : NOEMULT.AU

BF1 : 360.1300000 MHz

FW : 7200.00 Hz

INSTRUM : AM

NS : 178

O1 : 7201.32 Hz

O2 : 6596.29 Hz

SFO1 : 360.1372013 MHz

SW : 15.9583 ppm

SW_h : 5747.126 Hz

TD : 32768

TE : 298 K

*** Processing Parameters ***

FT_mod : fqr

OFFSET : 14.977 ppm

SF : 360.1346810 MHz

SI : 16384

*** 1D NMR Plot Parameters ***

SR : 4680.98 Hz

AQ_time : 2.8507960 sec

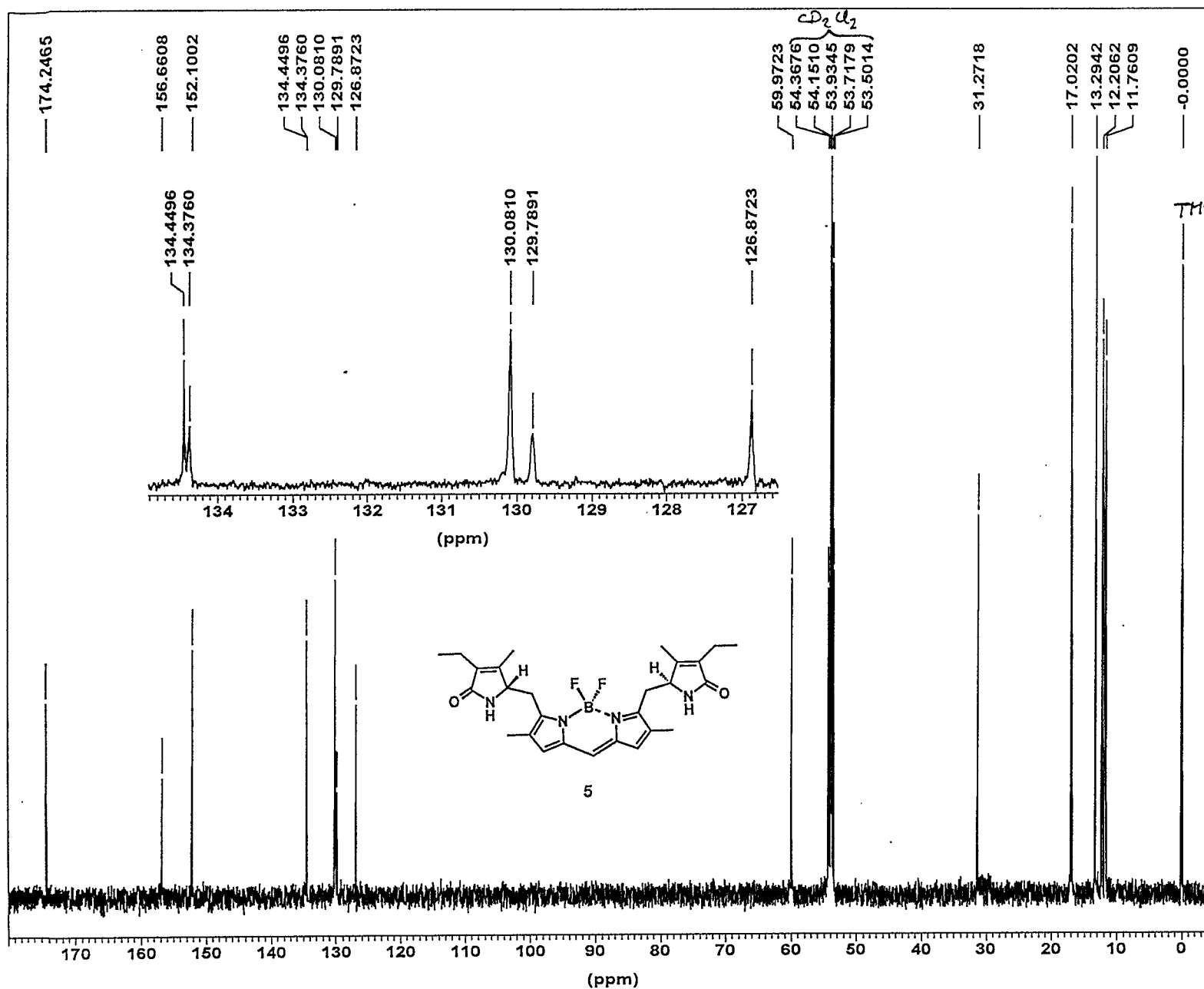
x ... Lösungsmittelpeak

¹³C-NMR, protonenentkoppelt (DRX500)

Probe: S,S-Urobilin als BF₂-Komplex

Lösungsm.: CD₂Cl₂ (TMS)

Compound 5: ¹³C-NMR (125.76 MHz) in CH₂Cl₂



*** Current Data Parameters ***

NAME : fehr7568.bbo
EXPNO : 2
PROCNO : 1

*** Acquisition Parameters ***

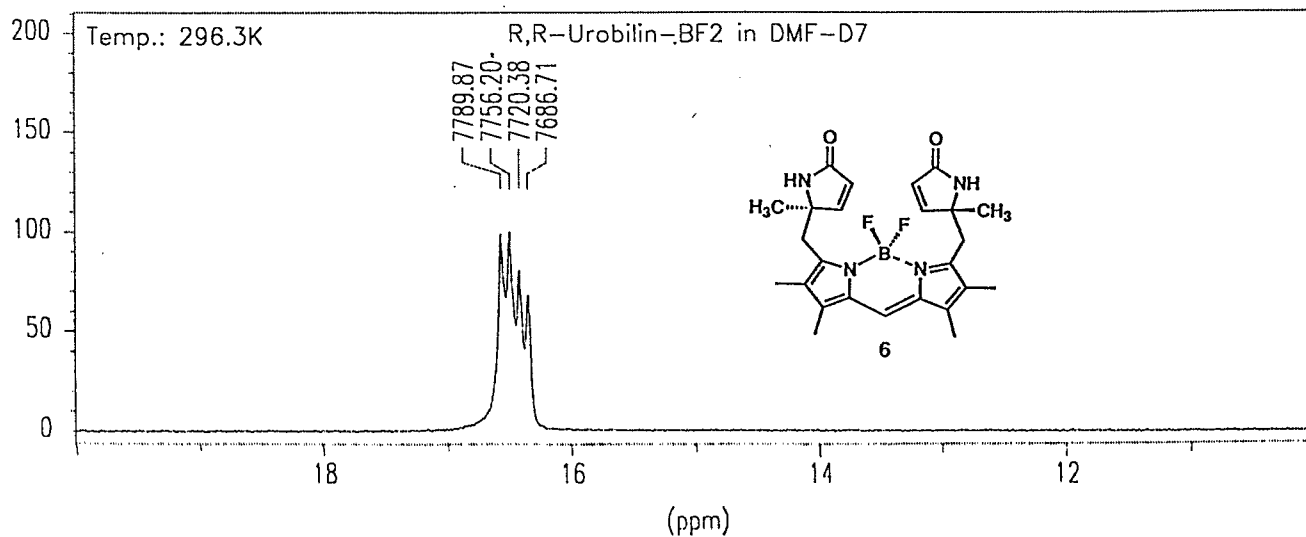
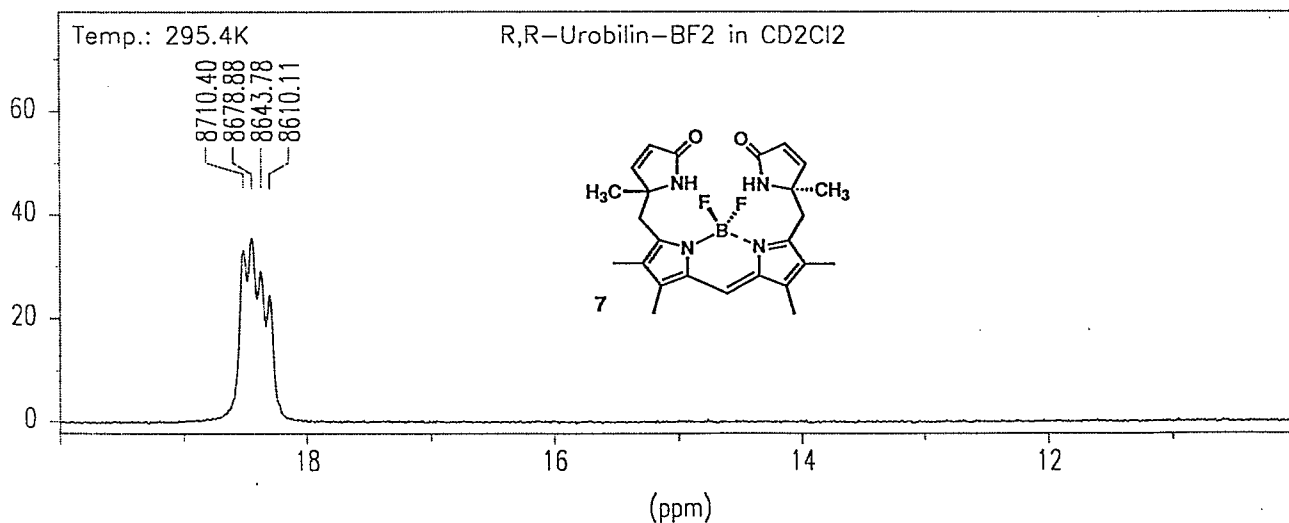
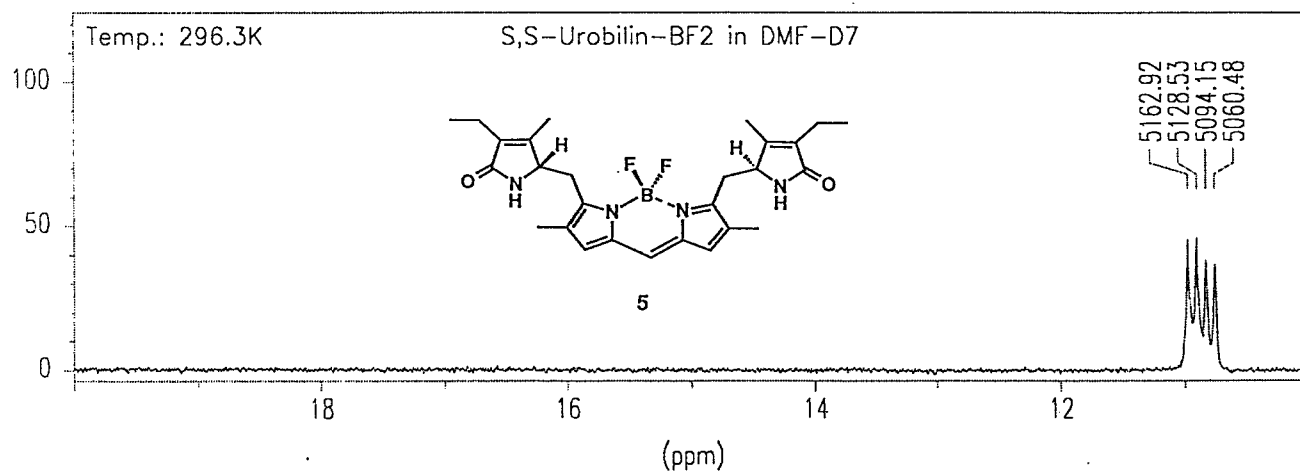
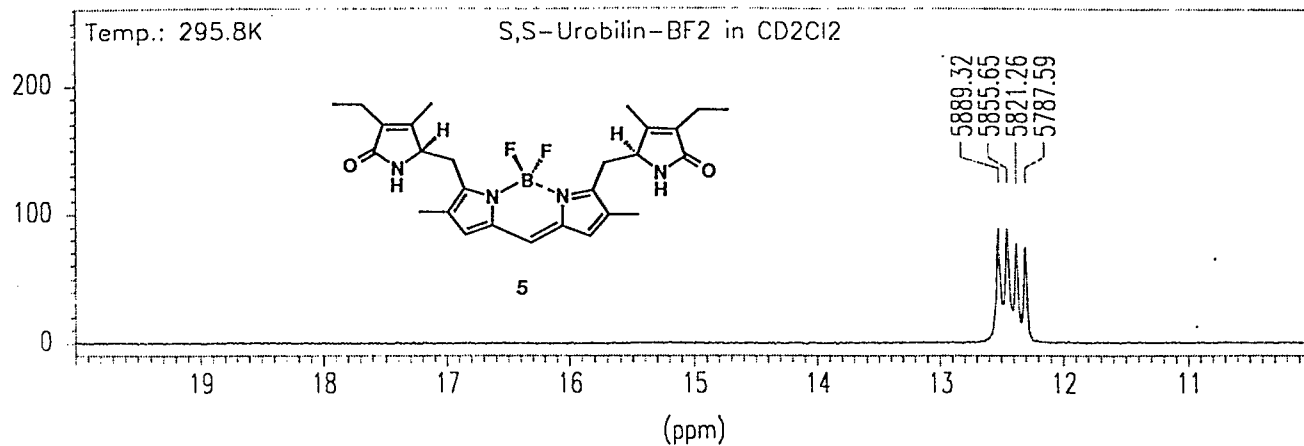
AQ_mod : qsim
AUNM : au_zg
BF1 : 125.7577390 MHz
DATE_d : Jul 04 1995
FW : 90000.00 Hz
INSTRUM : drx500
NS : 33674
O1 : 12867.09 Hz
O2 : 500.00 Hz
PARMODE : 1D
RG : 14596.5000000
SFO1 : 125.7706061 MHz
SW : 229.7972 ppm
SW_h : 28901.734 Hz
TD : 32768
TE : 283.0 K
V9 : 5.00 sec

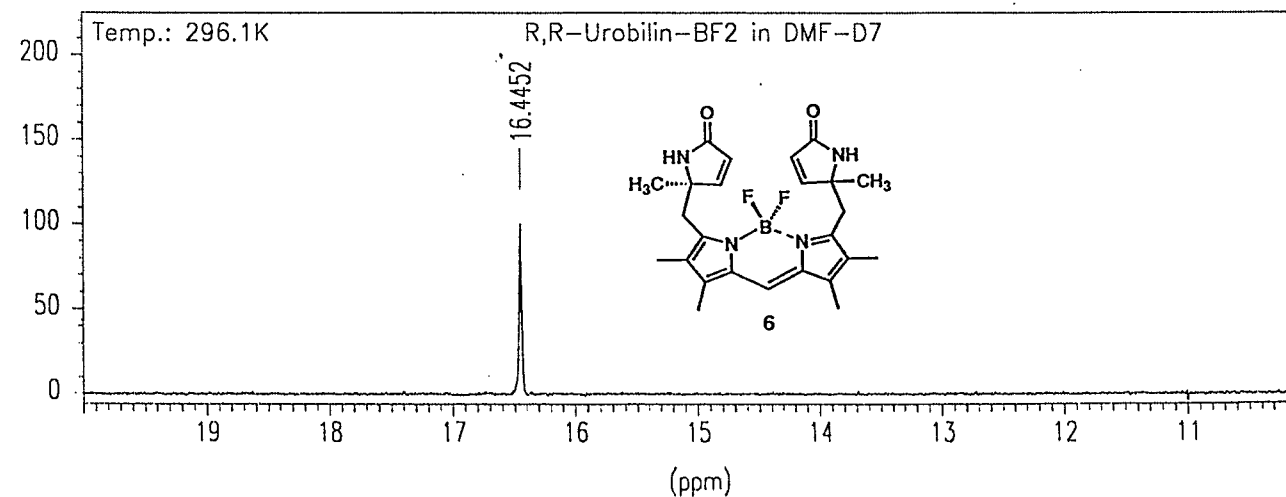
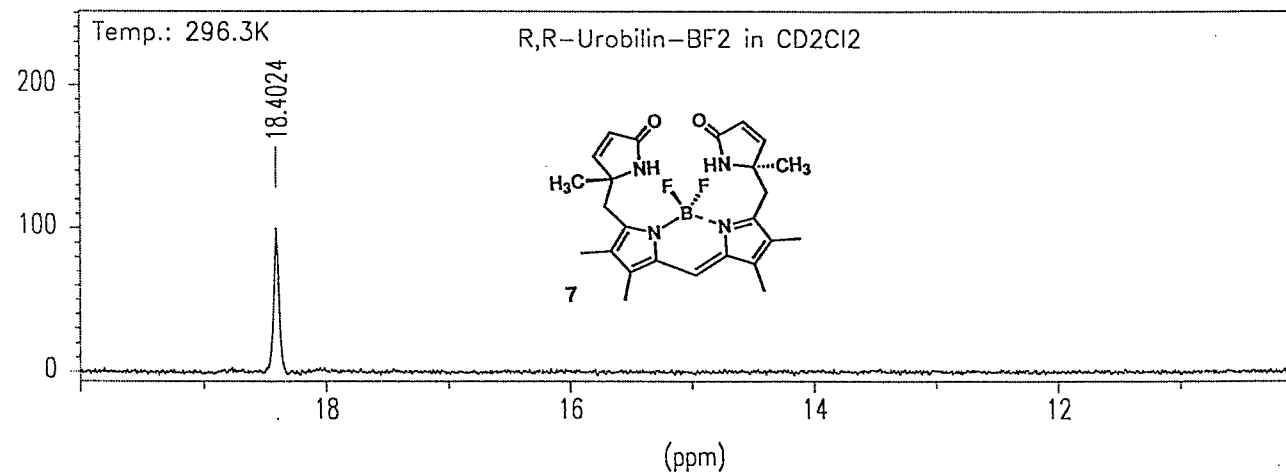
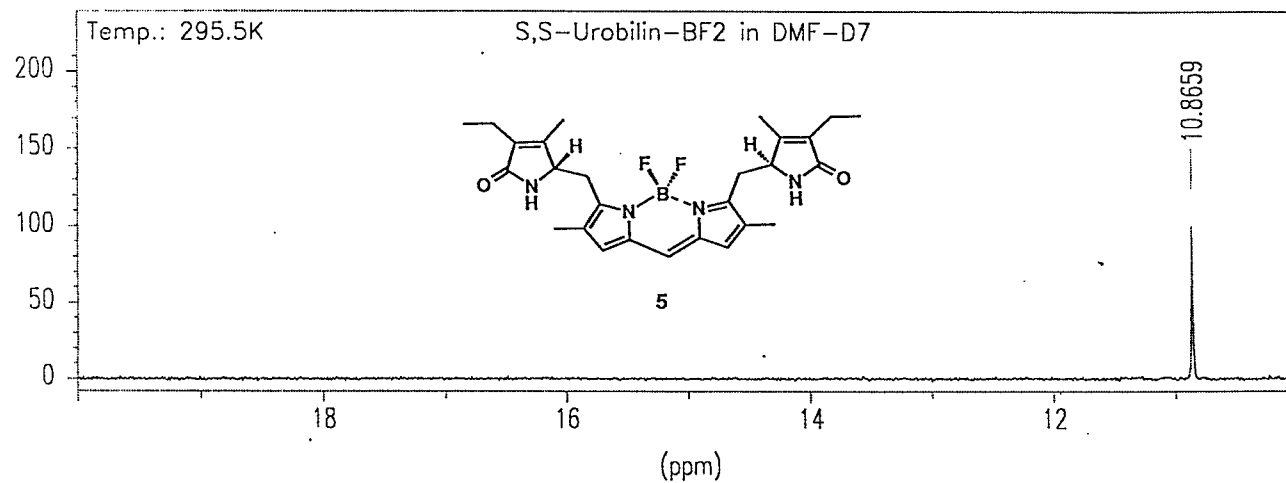
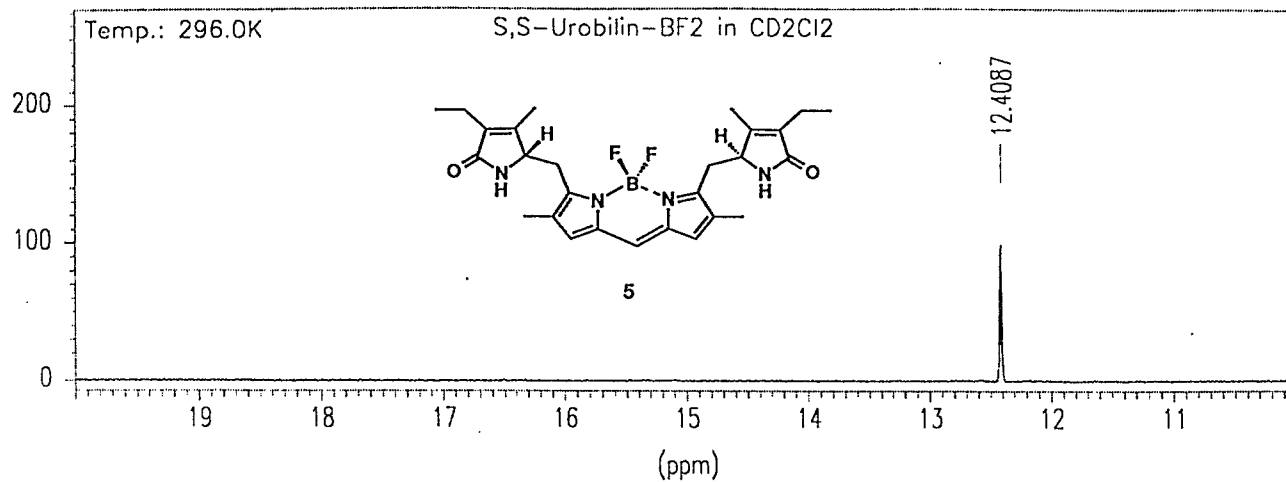
*** Processing Parameters ***

FT_mod : fqc
OFFSET : 220.040 ppm
SF : 125.7573853 MHz
SI : 262144

*** 1D NMR Plot Parameters ***

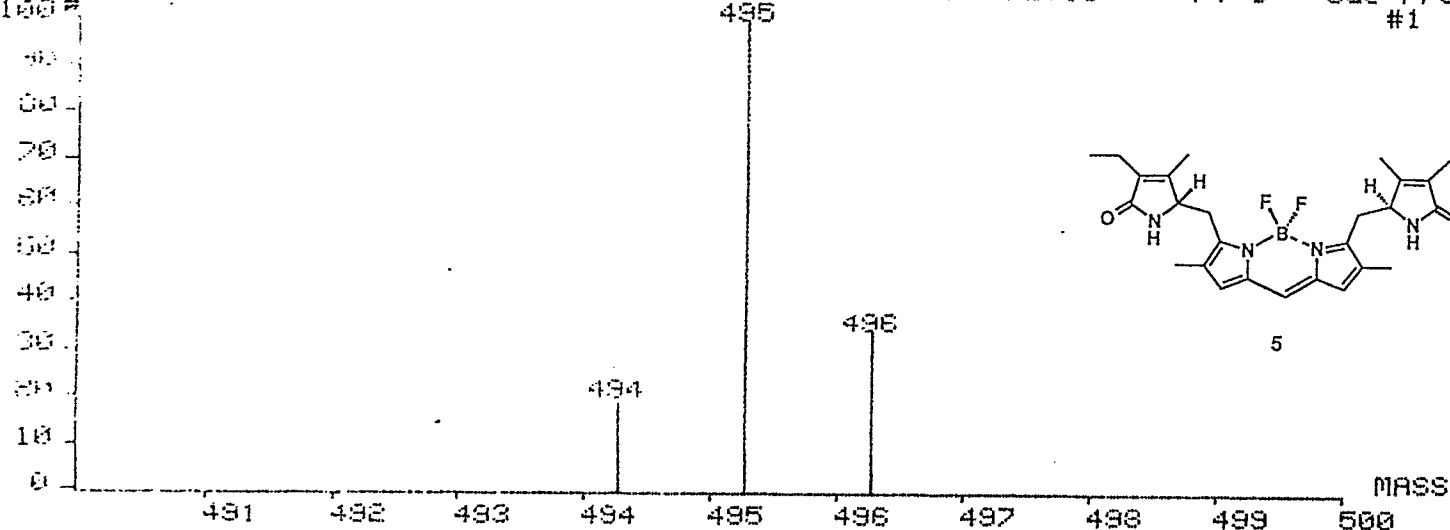
SR : -353.70 Hz
AQ_time : 0.5668860 sec





Compound 5: HR-FAB-MS

FEHR5552Y#1 x1 Bgd=0 20-APR-94 14:54+0:00:00 7070E FB+
 BpM=0 I=7.0v Hm=000 TIC=1078488064 RV Acnt:FRIBOURG Sys:FRB1
 Text:HR-FAB-MS: (S,S)-UROBILIN*BF2 [MATRIX: GLY./THIOGLY.] PT=0 Cal:FF01
 #1 3945000 1.0



File: FEHR5552Y

Scan #1

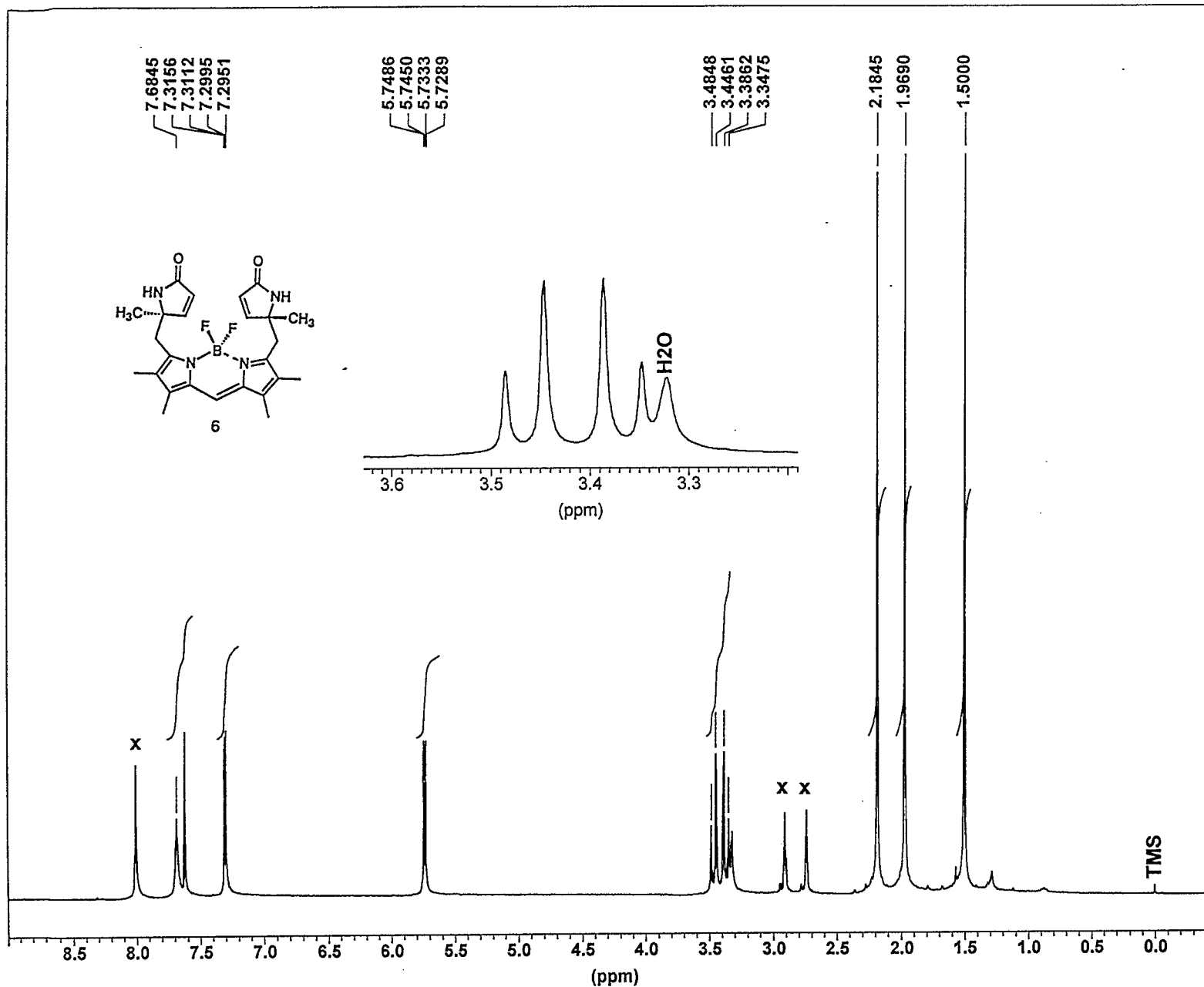
Page: 1-1

M/E	C	H	N	O	B	F	MMU	DBE	OBS. MASS	%INT
12										
495	30	33	4	1	1	1	-1.2	16.5	495.2743879	8.56
	27	31	4	1	1	3	-20.1	13.5		
	29	34	4	1	2	1	15.9	15.5		
	26	32	4	1	2	3	-3.0	12.5		
	27	34	4	2	1	2	-0.1	12.5		
	26	35	4	2	2	2	17.0	11.5		
	24	35	4	3	1	3	1.0	8.5		
	25	31	4	3	2	2	-19.4	12.5		
	26	33	4	4	1	1	-16.5	12.5		
	23	31	4	4	1	3	-35.3	9.5		
	25	34	4	4	2	1	0.6	11.5		
	23	34	4	5	1	2	-15.4	8.5		

Compound 6: ¹H-NMR (360.14 MHz) in DMF-*d*₇

Sample: R,R-Urobilin as BF₂-Complex

Solvent: DMF-d7 (TMS)



*** Current Data Parameter

NAME : NYDE7790

EXPNO : 1

PROCNO: 1

*** Acquisition Parameter

AQ_mod : qseq

AUNMNOEMULTI.AU

BF1	360.1300000 MHz
-----	-----------------

FW : 5400.00 Hz

INSTRUMENT AM

NS : 704

O1	:	7404.12 Hz
----	---	------------

O2 : 9554.00 Hz

SFO1 360.1374041 MHz

SW : 11.9687 ppm

SW_h : 4310.345 Hz

TD	:	32768
----	---	-------

TE : 312 K

*** Processing Parameters

FT_mod : no

OFFSET : 11.146 ppm

SF 360.1355453 MHz

SI : 16384

*** 1D NMR Plot Parameter

SR : 5545.34 Hz

AQ time : 3.8010680 sec

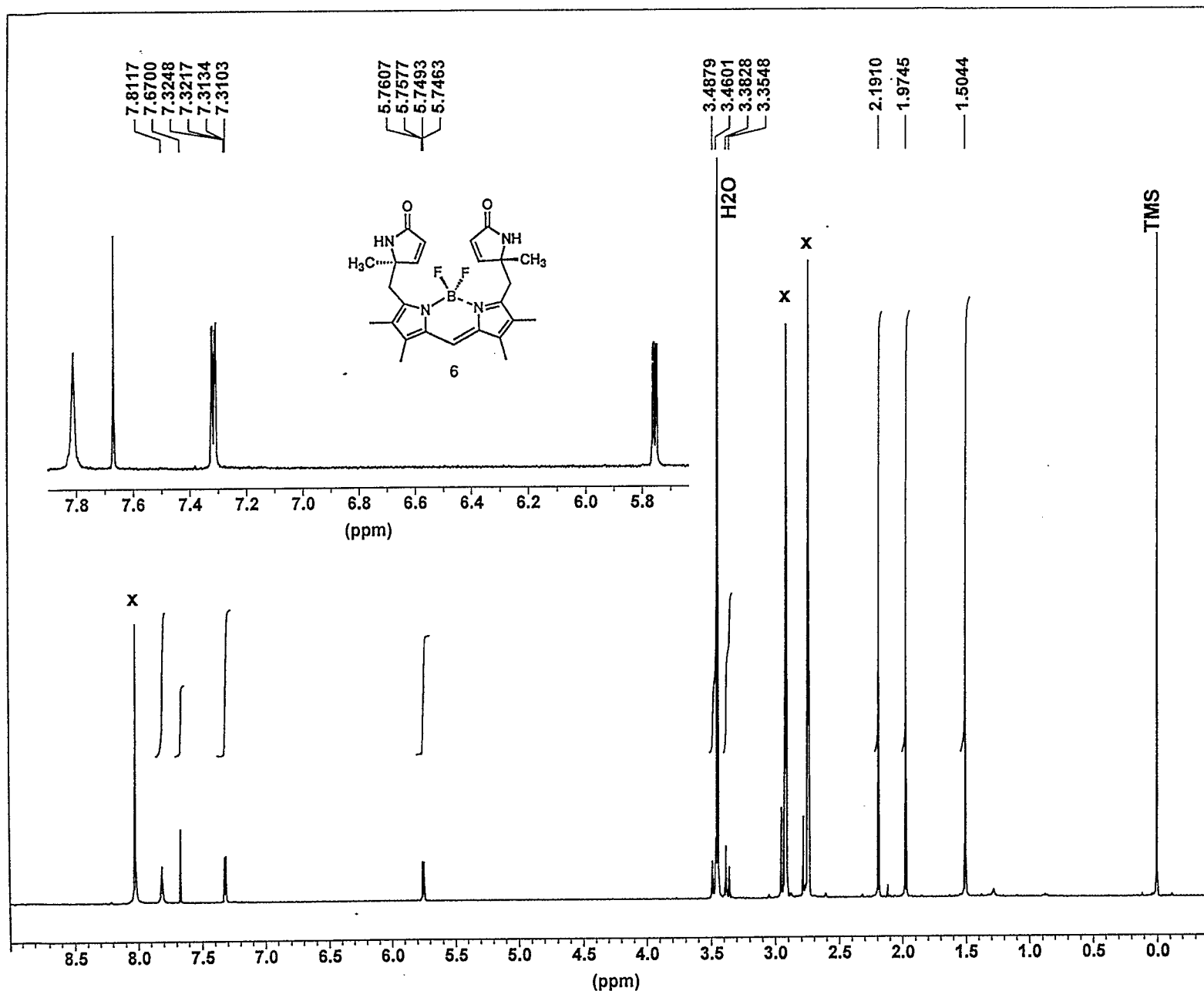
x ... solventpeak

¹H-NMR

Compound 6: ¹H-NMR (500.13 MHz) in DMF-d₇

Sample: R,R-Urobilin as BF₂-Complex

Solvent: DMF-d₇ (TMS)



*** Current Data Parameter

NAME nyde8956.bbo

EXPNO : 17

PROCNO: 1

*** Acquisition Parameter

AQ_mod : qsim

BF1 500.130000 MHz

DATE_d Aug 08 1995

FW : 90000.00 Hz

NS : 128

O1 : 4629.14 Hz

O2 : 0.00 Hz

PARMODE 1D

RG 812.7000122

SFO1 500.1346291 MHz

SW : 10.4794 ppm

SW_h : 5241.090 Hz

TD : 32768

TE : 299.6 K

V9 : 50.00 sec

AUNM :NOELT.AU

INSTRUM: drx500

*** Processing Parameters

FT_mod : fqc

OFFSET : 9.975 ppm

SF 500.1322608 MHz

SI : 131072

*** 1D NMR Plot Parameter

SR : 2260.79 Hz

AQ_time : 3.1260670 sec

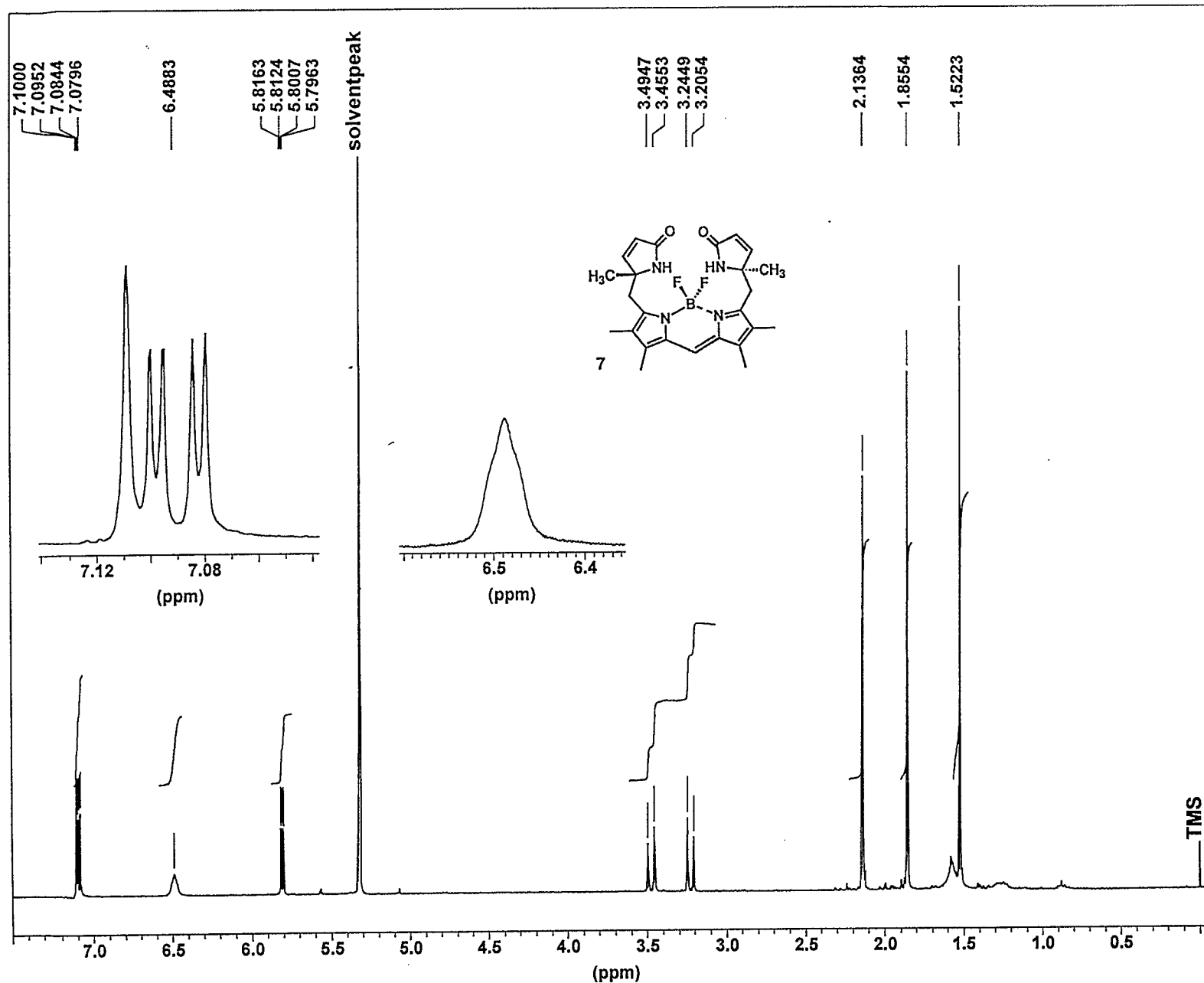
x ... solventpeak

¹H-NMR

Compound 7: ¹H-NMR (360.14 MHz) in CH₂Cl₂

Sample: R,R-Urobilin as BF₂-Complex

Solvent: CD₂Cl₂ (TMS)



*** Current Data Parameter

NAME : NYDE7762

EXPNO : 360

PROCNO: 1

*** Acquisition Parameter

AQ_mod : qseq

AUNMNOEMULTI.AU

BF1 360.1300000 MHz

FW : 3600.00 Hz

INSTRUM: AM

NS : 640

O1 : 6048.91 Hz

O2 : 6245.00 Hz

SFO1 360.1360489 MHz

SW : 7.9791 ppm

SW_h : 2873.563 Hz

TD : 32768

TE : 298 K

*** Processing Parameters

FT_mod : fqr

OFFSET : 7.789 ppm

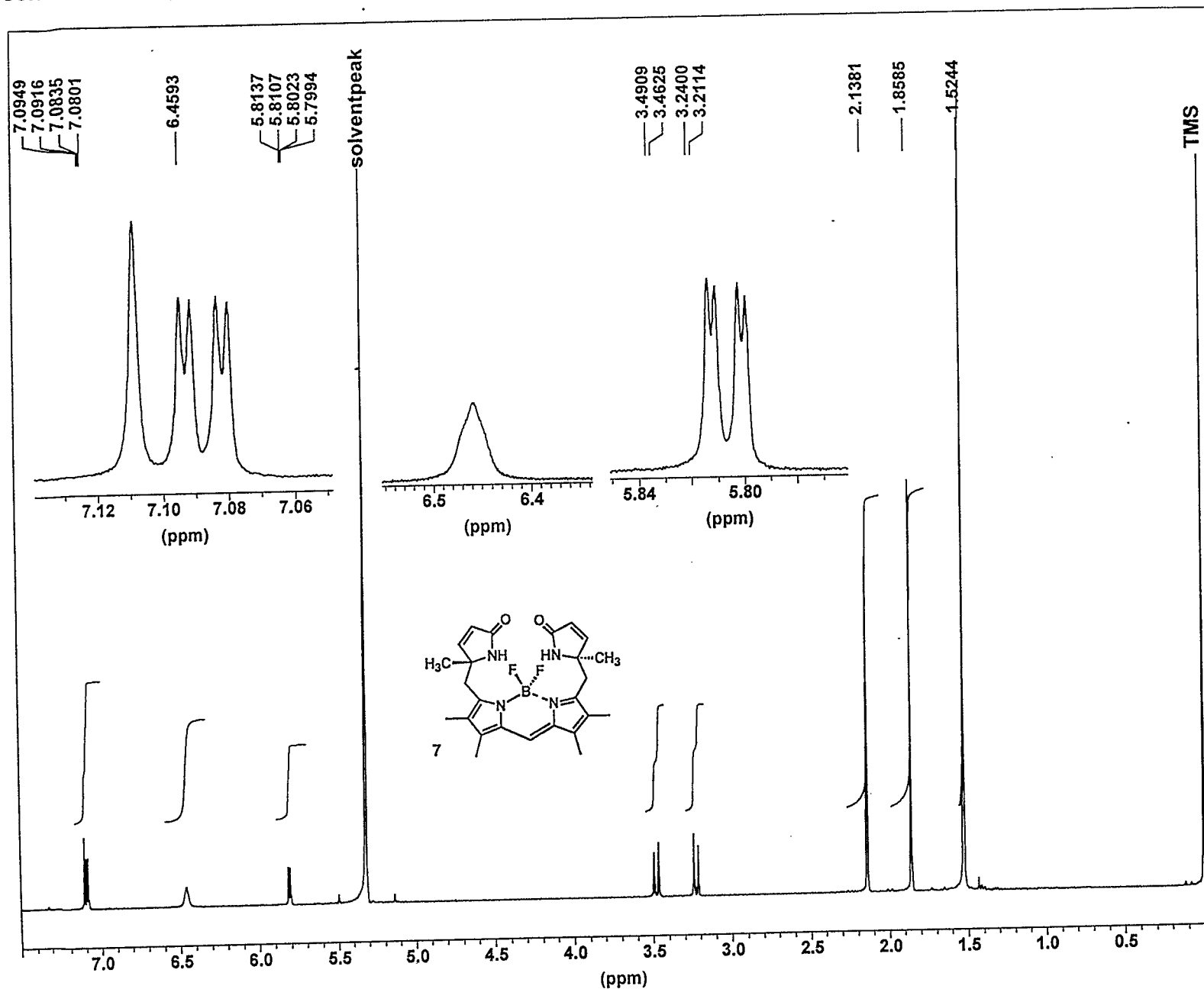
SF 360.1346805 MHz

SI : 16384

*** 1D NMR Plot Parameter

SR : 4680.56 Hz

AQ_time : 5.7016100 sec

1H-NMRSample: R,R-Urobilin as BF₂-ComplexSolvent: CD₂Cl₂ (TMS)**Compound 7: ¹H-NMR (500.13 MHz) in CH₂Cl₂******* Current Data Parameter**

NAME nyde8956.bbo

EXPNO : 10

PROCNO: 1

***** Acquisition Parameter**

AQ_mod : qsim

AUNM : NOELT.AU

BF1 500.1300000 MHz

DATE_d Aug 07 1995

FW : 90000.00 Hz

INSTRUM: drx500

NS : 128

O1 : 3357.77 Hz

O2 : 0.00 Hz

PARMODE 1D

RG 812.7000122

SFO1 500.1333578 MHz

SW : 10.4794 ppm

SW_h : 5241.090 Hz

TD : 32768

TE : 299.6 K

V9 : 50.00 sec

***** Processing Parameters**

FT_mod : fqc

OFFSET : 9.989 ppm

SF 500.1309825 MHz

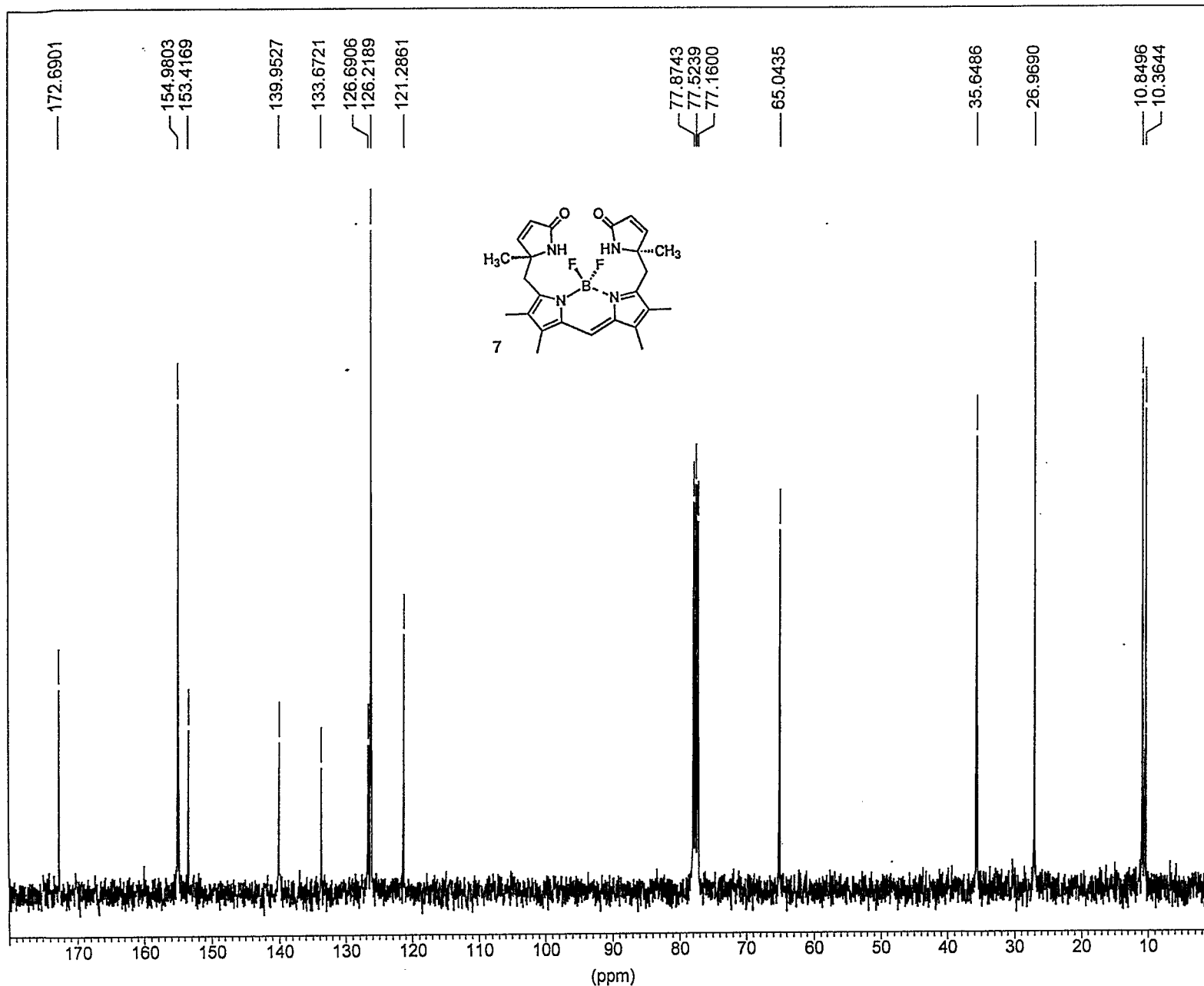
SI : 131072

***** 1D NMR Plot Parameter**

SR : 982.47 Hz

AQ_time : 3.1260670 sec

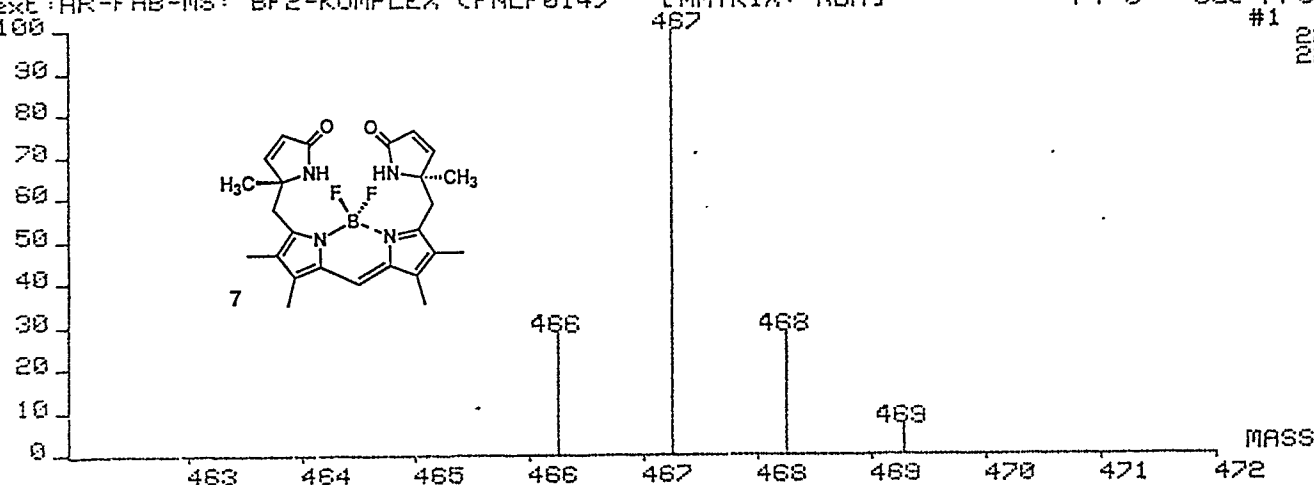
Solvent: CDCl₃



AQ_time : 0.8191130 sec

Compound 7: HR-FAB-MS

NYDE5652Y#1 x1 Bgd=0 10-JUN-95 08:51+0:00:00 7070E FB+
 SpM=0 I=10v Hm=500 TIC=2722045952 AY Acnt:FRIBOURG Sys:FAB1
 Text:HR-FAB-MS: BF2-KOMPLEX (FNLF014) [MATRIX: NBA] PT=0 Cal:FF01
 #1 22232000 1.0
 22232000



File: NYDE5652Y

Scan #1

Page: 1-1

calc.

$[M+H]^+$ $C_{25}H_{30}N_4O_2BF_2$
 467.242988

M/E	C	H	N	O	B	F	MMU	DBE	OBS. MASS	%INT
12										
467	28	29	4	1	1	1	-0.8	16.5	467.2426270	33.92
	26	34	4	1	1	2	36.7	11.5		
	25	27	4	1	1	3	-19.6	13.5		
	27	30	4	1	2	1	16.3	15.5		
	25	35	4	1	2	2	53.9	10.5		
	24	28	4	1	2	3	-2.5	12.5		
	27	25	4	2	1	1	-37.2	17.5		
	25	30	4	2	1	2	0.4	12.5		
	23	35	4	2	1	3	37.9	7.5		
	26	26	4	2	2	1	-20.0	16.5		
	24	31	4	2	2	2	17.5	11.5		
	25	33	4	3	1	1	20.3	11.5		
	24	26	4	3	1	2	-36.0	13.5		